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Effects of 2,4-D Formulation and Quinclorac on Spray Droplet Size and Deposition[1]

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Abstract: Studies were conducted on the campuses of Texas A&M University in College Station, TX, and New Mexico State in Las Cruces, NM, to determine the spray droplet size spectra produced by quinclorac and 2,4-D as the liquid, dry, and emulsion formulations during application with various nozzle sizes using a laser spectrometer. Quinclorac and 2,4-D formulations were also sprayed through three different nozzle sizes in a drift chamber and allowed to settle on glass slides placed downwind. The amounts of each herbicide deposited on the slides were quantified using high-performance liquid chromatography/photodiode array (HPLC/PDA) analysis to assess spray deposition of each formulation at different wind velocities. Data from the laser spectrometer suggested that formulations of 2,4-D affected droplet size, particularly when the 380 ml/min flat-fan nozzle was used. Quinclorac droplet sizes were similar to water regardless of nozzle size. Liquid and dry-formulated 2,4-D tended to be deposited downwind in greater quantities than the emulsion formulation when using the 380 and 760 ml/min spray nozzles with wind velocity of 15 km/h.

Nomenclature: 2,4-D, quinclorac.

Additional index words: Laser spectrometer, drift chamber.

Abbreviations: HPLC, high performance liquid chromatography; HSD, honestly significant difference; PDA, photodiode array.

INTRODUCTION

Spray drift is a constant concern for both agricultural producers and pesticide applicators. The problem of off-target injury associated with herbicide drift has been widespread since the beginning of herbicide use (Arle 1954). These problems continue in spite of prevention and mitigation efforts by producers, applicators, educators, cooperative extension, and legislators (Burn 2003; Texas Agriculture Code 1984; Ucar and Hall 2001).

The inverse relationship between droplet size and drift potential has been well documented (Derksen et al. 1999; Hobson et al. 1993). Larger droplet sizes are considered desirable for minimizing drift during pesticide applications, and nozzles have been designed specifically for producing such droplets over a range of conditions (Himel et al. 1990; Spraying Systems 1995). Although data regarding the effect of nozzle selection and pressure on droplet size have been collected, little information has been reported about the effects of herbicide formulation on spray atomization.

Emulsion formulations produce a more coarse droplet spectra than other formulations (Miller and Ellis 2000). Surfactants and other adjuvants are commonly added to spray mixtures to increase efficacy (Sharma and Singh 2001; Woznica et al. 2003), and researchers have found that the type and quantity of these adjuvants influence droplet size (Akesson et al. 1994; Apodaca et al. 1996; Mueller and Womac 1997).

Research has also been conducted to determine how chemical formulation and adjuvants affect droplet size with different spray mixtures (Hanks 1995; Yates and Akesson 1974) and with individual herbicides such as glyphosate (Mueller and Womac 1997) and propanil (Sanderson et al. 1997). However, it is not known how 2,4-D formulations affect droplet size or the likelihood of spray solution drift. This information would be valuable because 2,4-D, and to a lesser extent, quinclorac, have been linked to excessive drift and damage to non-target plants (Miller et al. 1963; Talbert et al. 2000).

Understanding the tendency of one formulation to form larger droplets than another could be valuable for reducing drift during application. In addition, informa-

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tion regarding the movement of spray particles containing specific herbicides would be useful. Therefore, the objectives of this research were to determine differences in droplet sizes of three formulations of 2,4-D and quinclorac using a laser spectrometer and to quantify differences among three 2,4-D formulations and between quinclorac and water in relation to the fate of droplet deposits and wind velocity.

MATERIALS AND METHODS

Laser Spectrometer. The experimental design was a completely randomized design with three 2,4-D formulations and water alone applied randomly through an 8001 (380 ml/min), 8002 (760 ml/min), and 8003 (1,140 ml/min) stainless steel flat-fan nozzles[3] typical of those used in various agricultural herbicide applications. Sprayer pressure was held constant at 280 kPa across all treatments. At this pressure, the 380, 760, and 1,140 ml/min nozzles emitted 0.4, 0.8, and 1.1 L/min, respectively.

The 2,4-D formulations included an emulsion containing the isooctyl (2-ethylhexyl) ester,[4] a prepackaged solution of the dimethylamine salt,[5] and a solution made from a dry crystal of the dimethylamine salt.[6] The only commercially available form of quinclorac was a dry formulation.[7] Herbicides were mixed in stainless steel containers and pressurized with CO_2 before treatment. The 2,4-D concentration was 2.8 mg/L and an application rate of 0.53 kg ae/ha, assuming a carrier rate of 190 L/ha. Treatments were mixed without supplemental surfactants or adjuvants, and each treatment was replicated three times.

Droplet size determination. Droplet size spectra were measured using a Malvern 2600c Laser Particle Size Analyzer[8] with an 800-mm lens. This laser spectrometer is one of several measurement systems used in many disciplines to measure particles in space (Kwong et al. 2000). The measurement process was automated so that when initiated, a carriage holding the nozzle was moved through a laser beam. As the spray plume entered the beam, a detector recorded the different light diffractions caused by the varying sizes of spray particles moving through the light. The software then converted these diffraction measurements to droplet size results (Rawle 1995). An example of data distribution is provided in Figure 1.

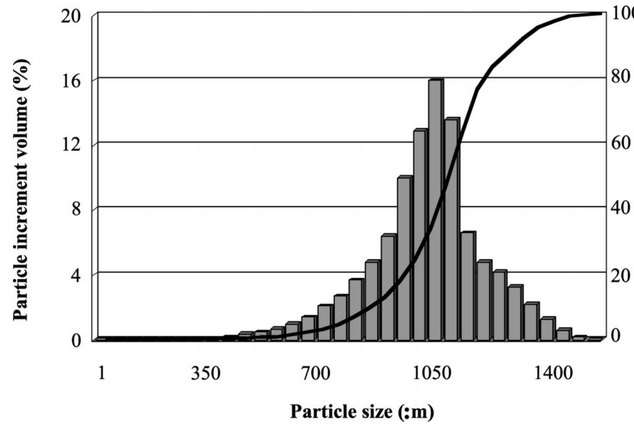

Figure 1. Typical laser spectrometer output containing droplet size spectrum. Bars represent the amount of the spray volume that was contained in each size category, and the solid line is the amount of spray volume less than that size. These data represent the spectrum from water sprayed through a 760 ml/min nozzle.

The laser spectrometer records droplet sizes as small as 5 μm and as large as 1,500 μm, but the smaller droplets are critical in assessing physical drift. Previous investigations have shown the droplet size threshold of 150 to 200 μm is critical in reducing particle movement during application (Bode 1987). Drops larger than this threshold exhibit limited movement because of their shorter fall-time. Therefore, only droplet sizes smaller than 191 μm will be discussed.

Results for 2,4-D and quinclorac are presented as volume median diameter ($D_{v0.5}$) values, which represent the median droplet size by volume, and the percent volume contained in droplets less than 191 μm. All results were arranged by nozzle because nozzle comparison was not a priority goal of this work.

Results were subjected to ANOVA, and means for 2,4-D formulations were separated using Tukey's Honestly Significant Differences (HSD) procedure at $P \leq 0.05$ (SAS 1985). Only one quinclorac formulation was used, so the means of quinclorac treatments were compared with water alone using two-tailed *t* tests at $P \leq 0.05$ with SPSS.[9]

Herbicide Deposition. *Chamber description.* The drift chamber was designed to move spray particles emitted from the nozzle at the intake-end of the duct to glass slides placed downwind for collection. The chamber consisted of a square sheet-metal duct, a nozzle assem-

[3] Spray nozzles. Spraying Systems Co., Wheaton, IL, 60188.
[4] Low Vol® herbicide. Platte Chemical Co., Greeley, CO, 80631-5852.
[5] Weedar 64® herbicide. Rhone-Poulenc Ag Company, Research Triangle Park, NC 27709.
[6] Savage® herbicide. Platte Chemical Co., Fremont, NE 68025-5697.
[7] Facet® herbicide. BASF Corporation, Florham Park, NJ 07932.
[8] Particle size analyzer. Malvern Instruments, Malvern, U.K. WR14 1XZ.
[9] Statistical Package for the Social Sciences, SPSS Inc., 233 S. Wacker Drive, 11th floor, Chicago, IL 60606.

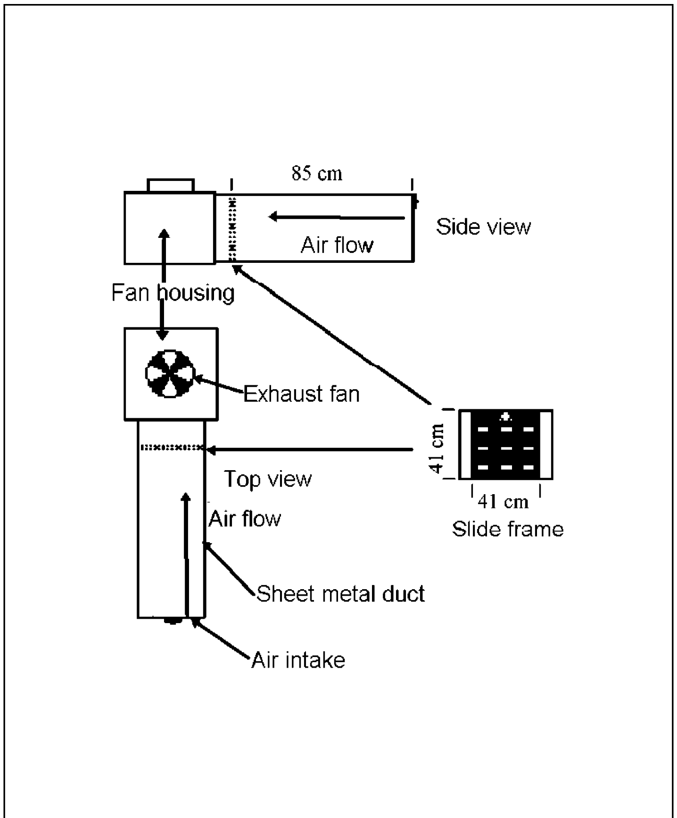

Figure 2. Top and side view of the spray chamber used to direct spray particles toward glass slides in the deposition study and a front view of the slide rack that shows placement of slides for catching spray droplets.

bly, and a fan housing containing the fan and electric motor (Figure 2). The duct was 119 cm long and 41 cm wide by 41 cm high. The nozzle assembly was attached to the top of the sheet-metal duct 2.5 cm away from the opening at a height of 40 cm from the base of the housing. A rheostat controller was added to the motor so that the wind velocity could be adjusted. Wind velocities were monitored using a hot-wire anemometer[10] placed at the intake end of the duct. Exhaust from the drift chamber was removed from the building by a high-volume ventilation fan.

A steel frame was constructed to hold glass slides[11] (25 by 75 by 1 mm) inside the drift-chamber duct. Glass slides were placed perpendicular to the air flow at three heights inside the duct. Each height level contained three slides 85 cm from the nozzle and spaced evenly across the width of the duct to allow air to flow (Figure 2). The slides in the lowest, middle, and top heights were 5, 20, and 35 cm from the duct floor, respectively.

2,4-D. This study was designed as a three by three factorial experiment with deposition from three 2,4-D formulations being quantified at three heights downwind. The 2,4-D formulations used were the same as those used in the laser spectrometer experiment. Each formulation was applied through 380, 760, and 1,140 ml/min nozzles at three wind velocities of 3, 7, and 15 km/h. Herbicides were mixed without surfactants in 3-L containers and pressurized with CO_2 before treatment. The CO_2 pressure was maintained at 280 kPa throughout the experiment. The 2,4-D concentration was equivalent to 0.53 kg/ha assuming a carrier rate of 190 L/ha.

Nine glass slides were positioned in the slide frame inside the duct before each treatment. With the wind velocity held constant at 3, 7, or 15 km/h, a 2-s emission was sprayed through the nozzle at the duct intake. The three slides from each height level were carefully collected and placed into one vial containing 7 ml of methanol. Any amount of 2,4-D lost through volatilization during the experimental procedure was considered negligible. The slide frame was washed with methanol and dried with compressed air before each treatment. This process was repeated for each herbicide with every nozzle and wind velocity.

A 1-ml aliquot was removed from each collection vial and placed in a 1-ml glass sample vial so that the amount of herbicide captured could be determined through HPLC analysis. Results were subjected to ANOVA using SPSS statistical software, and means were separated using Tukey's HSD at $P \leq 0.05$.

Quinclorac. The quinclorac treatments were randomized for each nozzle and wind velocity. The study was carried out identical to the 2,4-D study except that the quinclorac treatments were compared with water. The quinclorac concentration used was equivalent to 0.56 kg/ha assuming a carrier rate of 190 L/ha. Results were subjected to ANOVA, and concentration means were separated by Tukey's HSD at the 5% level of significance using SPSS statistical software.

Quantitation. The amounts of 2,4-D or quinclorac captured at each slide level were determined using HPLC with a PDA detector.[12] The column used for detection was a Waters Symmetry Shield RP_8[12] (3.5 μm, 2.1 mm interior diameter [I.D.] by 150 mm) that was maintained at 21 C during analysis. Data from the analysis was processed with Millennium[32] Chromatography Manager software.[12] A 10-μl sample of methanol was injected from each of the glass sample vials collected during the

[10] Hot-wire anemometer. Fisher Scientific. Pittsburg, PA 15275.
[11] Glass slides. VWR Scientific Inc. West Chester, PA 19380.

[12] Waters Corporation, 34 Maple Street, Milford, MA 01757.

Table 1. Volume median diameter and percent (%) spray volumes less than 191 μm detected by a laser spectrometer of four spray mixtures through 380, 760, and 1,140 ml/min spray nozzles.

Spray mixture	Volume median diameter			Percent spray volume <91μm		
	Nozzle flow[a]			Nozzle flow		
	380 ml/min	760 ml/min	1,140 ml/min	380 ml/min	760 ml/min	1,140 ml/min
	$D_{v\,0.5}$			%		
Emulsion	318 a	360 a	381 a	15.2 a	11.3 a	11.2 a
Liquid	314 a	352 ab	357 ab	14.8 a	13.2 ab	18.0 b
Dry	305 b	344 bc	350 bc	20.5 b	14.8 bc	20.3 b
Water	302 b	335 c	364 c	20.7 b	16.7 c	17.9 b

[a] Means with identical letters within columns are not different at P ≤ 0.05 according to Tukey's Honestly Significant Differnce (HSD).

study. All injections were performed automatically with a Waters 717 autosampler.[12]

Samples containing the 2,4-D salt and ester formulations were analyzed at wavelengths of 198.4 and 199.2 μm, respectively. Quinclorac samples were analyzed at 224.8 μm. These optimal wavelength values were determined by studying the three-dimensional absorbance graphs produced during HPLC method development procedures (data not shown). Samples containing quinclorac and 2,4-D from the liquid and dry treatments were analyzed using a mobile phase of 50% acetonitrile[13] and 50% water buffered to pH 3.4 with sodium phosphate[13] (NaH_2PO_4:H_2O) and phosphoric acid.[13] Samples containing 2,4-D from the emulsion formulation were analyzed using 70% acetonitrile and 30% water buffered to pH 3.4 with sodium phosphate and phosphoric acid. Separate analysis of the emulsion formulation was necessary because it contained an ester form of 2,4-D whereas the liquid and dry had the dimethylamine salt.

RESULTS AND DISCUSSION

2,4-D Droplet Size Spectra. *380 ml/min nozzle.* The $D_{v0.5}$ for the liquid and emulsion formulations were not different from each other and were larger than that of the dry formulation when sprayed through the 380 ml/min nozzle (Table 1). The $D_{v0.5}$ values of the dry formulation were similar to that of water alone. Similarly, the emulsion and liquid 2,4-D formulations produced significantly less volume in droplet sizes (<191 μm) than the dry formulation or water alone (Table 1). The spray volume <191 μm produced by the dry formulation was similar to that of water alone.

760 ml/min nozzle. As was seen with the 380 ml/min nozzle, the liquid and emulsion 2,4-D formulations produced $D_{v0.5}$ values that were similar to each other (Table 1). The similarity found between the dry formulation and water alone passing through the 380 ml/min nozzle was also present when these formulations were sprayed through the 760 ml/min nozzle. However, dry formulation $D_{v0.5}$ values were comparable to those from the liquid formulation. Similarly, the emulsion and liquid formulations produced less volume in droplet sizes <191 μm than water alone (Table 1). The volumes <191 μm for the dry formulation and water alone were similar. However, similarity between spray volumes <191 μm from the liquid and dry was also detected.

1,140 ml/min nozzle. The emulsion produced larger $D_{v0.5}$ values than any other 2,4-D formulation when sprayed through the 1,140 ml/min nozzle (Table 1). The liquid and dry formulations were similar to each other and water alone. The emulsion also produced less spray volume <191 μm than the other 2,4-D formulations (Table 1). Water, liquid, and dry formulations were not different from each other. The similarity between the liquid and dry formulation recorded with the 760 and 1,140 ml/min nozzles but not with the 380 ml/min nozzle suggests that the effect of herbicide formulation on droplet size decreased as nozzle size increased.

Although droplet-size increases brought about by changing formulations within each nozzle were at times statistically significant, they were not of the same magnitude that was noted by increasing nozzle size. When the data were averaged within each formulation, the increase in $D_{v0.5}$ values from the dry to emulsion formulation was 9.0%. In contrast, when data were averaged within each nozzle, the $D_{v0.5}$ from the 1,140 ml/min nozzle was 17.1% larger than that from the 380 ml/min nozzle. This information suggests that whereas the formulation of 2,4-D affects spray droplet size, nozzle selection is likely more influential in reducing drift during application.

Quinclorac Droplet Size Spectra. No differences were detected between the $D_{v0.5}$ values of the quinclorac spray solutions and that of water for any nozzle (Table 2). Sim-

[13] EM Science, 480 S. Democrat Road, Gibbstown, NJ 08027.

Table 2. Main effects of quinclorac on the median diameters of spray droplets and spray volume ł91 μm droplet size.

Nozzle flow[a]	Main effect	Test statistic	Degrees of freedom	Probability value <0.05
ml/min				
380	$D_{v0.5}$	0.44	4	0.68
	% volume <191 μm	0.18	4	0.87
760	$D_{v0.5}$	0.30	4	0.78
	% volume <191 μm	0.79	4	0.47
1,140	$D_{v0.5}$	1.10	4	0.33
	% volume <191 μm	1.05	4	0.35

[a] Nozzle code denotes relative orifice size and spray angle of the nozzles used in the study.

ilarly, there were no differences between quinclorac and water for spray-droplet volumes <191 μm. This response suggests that the spectra of quinclorac spray solution through these nozzles are not different from that of water alone.

2,4-D Deposition. *3 km/h wind velocity.* No differences were detected among the amounts of 2,4-D deposited on the glass slides at any height using the 380 or 760 ml/min nozzles in the 3 km/h wind (Table 3). When sprayed through the 1,140 ml/min nozzle, the liquid formulation was present in greater concentrations than the emulsion at all three deposition heights. The dry formulation was deposited in concentrations up to 67% lower than the liquid, but these differences were not significant. Similarly, the emulsion formulation was deposited on the top height of slides in concentrations up to 78% lower than the dry, but these differences were also insignificant.

7 km/h wind velocity. There were no differences among the concentrations of 2,4-D deposited at any height using the 380 or 1,140 ml/min nozzles in the 7 km/h wind (Table 3). The concentration of the liquid formulation was greater than that of the emulsion at the highest height when sprayed through the 760 ml/min nozzle. However, this pattern was not observed on the midheight slides with that nozzle where the dry formulation was present in greater quantities than the emulsion. The slides at the lowest height of the 760 ml/min nozzle treatments contained the liquid formulation in greater quantities than the dry and emulsion.

15 km/h wind velocity. There were no differences among the concentrations of the three 2,4-D formulations deposited at the highest height when using the 380 and 760 ml/min nozzles in a 15 km/h wind (Table 3). However, the liquid formulation concentration was greater at midheight than the emulsion with each of these nozzles. Likewise, the emulsion concentration was less than that of the other two formulations at the lowest height with the 380 and 760 ml/min nozzles. There were no differences among 2,4-D concentrations from the three formulations at the top or midheight slides when using the 1,140 ml/min nozzle in the 15 km/h wind. The lowest height contained 2,4-D from the dry formulation in greater quantities than that recorded for the emulsion.

The 2,4-D concentrations from the emulsion formulation were among the lowest in five of the nine treatments in 15-km/h wind (Table 3), suggesting that the

Table 3. Amounts of emulsion, liquid, and dry 2,4-D formulations captured in a 3-, 7-, and 15-km/h wind using glass slides placed at three heights downwind of a spray nozzle.

		Wind velocity[a] (km/h)								
		3 Nozzle flow[b]			7 Nozzle flow			15 Nozzle flow		
Slide height (cm)[c]	Spray mixture[d]	380[e]	760	1,140	380	760	1,140	380	760	1,140
		ml/min								
5	Emulsion	0 a	0 a	1.5 b	9.0 a	7.4 a	14.5 a	21.2 b	36.1 b	25.9 b
	Liquid	4.4 a	4.4 a	9.5 a	42.6 a	82.1 a	24.6 a	140.4 a	154.9 a	105.9 ab
	Dry	2.5 a	3.5 a	3.1 ab	13.9 a	29.3 a	19.2 a	136.5 a	202.7 a	188.8 a
20	Emulsion	0 a	3.8 a	0.5 ab	5.3 a	9.2 a	26.5 a	7.3 b	23.6 b	10.3 a
	Liquid	3.9 a	4.6 a	7.4 a	19.4 a	29.6 a	21.9 a	69.8 a	102.0 a	33.3 a
	Dry	5.3 a	3.5 a	2.2 b	12.7 a	35.3 a	25.5 a	41.9 ab	76.1 ab	56.7 a
35	Emulsion	0 a	3.7 a	0.5 b	0.5 a	0.7 b	11.3 a	4.1 a	11.6 a	1.9 a
	Liquid	5.3 a	9.6 a	7.8 a	22.5 a	22.5 a	9.0 a	10.4 a	14.9 a	29.9 a
	Dry	2.3 a	5.6 a	2.6 ab	19.2 a	19.2 ab	23.2 a	6.9 a	32.7 a	31.4 a

[a] Wind velocity: 3-, 7-, and 15-km/h wind velocities simulated through a drift chamber.

[b] Nozzle size: Spray mixtures were applied through 380-, 760-, 1,140-ml/min spray nozzles. The codes represent the spray angle and relative output of the nozzles used in the experiment.

[c] Slide height: Distances were measured from the duct floor to the top of the glass slide.

[d] Spray mixture: Emulsion contained the isooctyl (2-ethylhexyl)ester of 2,4-D; liquid, contained a premade solution of the dimethylamine salt of 2,4-D; dry, contained a solution made from the dry crystal of the dimethyl amine salt of 2,4-D.

[e] Means within each column and slide height were separated using Tukey's Honestly Significant Differnce (HSD) (0.05).

Table 4. Amount of quinclorac captured on glass slides placed at three heights downwind of a spray nozzle in a 3-, 7-, and 15-km/h wind through a drift chamber.[a]

Wind velocity	Slide height[b]	Quinclorac captured
km/h	cm	μg
3	35	0.8 a
	20	5.2 b
	5	8.9 c
7	35	20.1 a
	20	24.3 a
	5	33.2 b
15	35	18.4 a
	20	70.9 a
	5	146.0 b

[a] Means of quinclorac captured with identical letters within columns are not different at the 5% significance level according to Tukey's Honestly Significant Differnce (HSD).

[b] Slide height: Distances were measured from the duct floor to the top of the glass slide.

emulsion formulation was least likely to drift off-target during application under these conditions. This finding coincides with data collected using the laser spectrometer, as the $D_{v0.5}$ and spray volume <191 μm from the emulsion indicated droplets that would resist movement.

In contrast, the 2,4-D concentrations collected from the liquid formulation were among the highest in five of the nine 15-km/h wind treatments (Table 3), which is in contrast to the spectrometer data for that formulation. This finding suggests that the liquid formulation is susceptible to movement under high wind conditions in spite of the larger droplet size.

Misleading results brought about by volatilization of the 2,4-D ester from the deposited spray droplets are unlikely here, as the glass slides were quickly placed into methanol after each treatment. Using this experimental procedure, the slides were not allowed to dry and there was little time for volatilization to occur. The droplets from the emulsion could be more dense with a higher specific gravity than those from the liquid, which would explain the contrasts in deposition of those formulations even though their sizes are similar.

Quinclorac Deposition. Slides in the top position captured the least amount of quinclorac when the wind velocity was 3 km/h, whereas the low position captured the most quinclorac (Table 4). When the wind velocity was increased to 7 km/h, the lowest slide position again captured the greatest amount of quinclorac. Slides at the medium and high heights captured less quinclorac than those in the lowest position. In the 15-km/h wind, the lowest slide position captured the greatest amount of quinclorac. The high and medium slide positions were not different from each other and captured less than the slides in the lowest position.

An increase in quinclorac concentration of greater than 16× from the low height in 3 km/h wind to the low height in 15 km/h wind suggests that quinclorac is susceptible to movement during application under these windy conditions (Table 4). Because no other form of quinclorac was available, future research should focus on other methods of reducing quinclorac drift such as the addition of drift-reducing agents to the spray mixture.

The dry formulation of 2,4-D and quinclorac were similar in that their spray volumes in droplets < 191 μm and $D_{v0.5}$ values were not different than water alone throughout the study. This finding could indicate similarities in droplet formation with herbicides formulated as dry products. The ionic properties of these products could affect droplet sizes during herbicide application. Future research is needed to investigate the ionic properties of dry-formulated herbicides and to determine what effect this has on droplet size.

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